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(54) Process for dispersing ignition resistant additives into carbonate polymers

Verfahren zur Verteilung von entzündungsfesten Zusätzen in Carbonatpolymeren

Procédé de dispersion d'additifs de résistance à l'inflammation dans des polycarbonates

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(72) Inventor: **Ogoe, Samuel Abotar**
Missouri City, Texas 77459 (US)

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(74) Representative: **Casalonga, Axel et al**
D-80469 München (DE)

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EP-A- 0 000 186 EP-A- 0 046 558
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(73) Proprietor: **THE DOW CHEMICAL COMPANY**
Midland, Michigan 48640-1967 (US)

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Description

This invention relates to a process for the production of ignition resistant or flame retardant polycarbonates and/or carbonate polymers wherein the ignition resistant additives are first compounded into a polycarbonate carrier resin then blended with a low molecular weight or moldable polycarbonate.

It is known from US-A-4,626,563 that aromatic sulfimide metal salts, monomeric or polymeric halogenated aromatic compounds, metal salts of inorganic acids having a pKa of from 1 to 5, and fibril-forming polytetrafluoroethylene are useful to render carbonate polymers ignition resistant or flame retardant. Other flame retardant additives are known from and listed in US-A-4,650,823. These patents are incorporated by reference herein.

The present invention is broadly directed to a process for dispersing one or more powdered ignition resistant or flame retardant additives into carbonate polymers wherein the impact properties of the ignition resistant carbonate polymers is improved.

More specifically, the invention is directed to a process for dispersing one or more powdered ignition resistant or flame retardant additives into carbonate polymers to make polymers having an embrittlement of less than 50% which comprises the steps of preparing a base concentrate having from 5 to 50 weight per cent based on concentrate of said additives by melt blending said additives with a carbonate polymer having a melt flow rate (MFR) of from 3 to less than 40 and preferably a MFR in the range of from 3 to 20, said additives being selected from the group of :

- i) a metal salt of an organic aromatic sulfur containing compound,
- ii) a monomeric, oligomeric or polymeric halogenated aromatic compound,
- iii) a metal salt of an inorganic compound
- iv) a free aromatic sulfimide, and
- v) a fibril forming polytetrafluoroethylene,

pelletizing said base concentrate having said ignition resistant additive dispersed therein, dry blending said concentrate pellets with carbonate polymer pellets having a melt flow rate of from 1 to less than 40 whereby said polymer/concentrate blend has an amount of said additive dispersed therein effective to render said blend ignition resistant, and pelletizing or molding said carbonate polymer/concentrate blend.

The preferred process of this invention has the following steps:

A) preparing a base concentrate by blending a carbonate polymer having a melt flow rate in the range of from 3 to less than 40 with an additive composition comprising

- i) 2.0 to 10 percent by weight of a metal salt of an organic aromatic sulfur containing compound,
- ii) 10 to 80 percent by weight of a monomeric, oligomeric or polymeric halogenated aromatic compound,
- iii) 2.0 to 10.0 percent by weight of a compound selected from a metal salt of an inorganic compound and a free aromatic sulfimide, and
- iv) 6 to 40 percent by weight of a fibril-forming polytetrafluoroethylene;

B) blending said base concentrate pellets with carbonate polymer pellets having a melt flow rate of from 1 to less than 40 and substantially lower than that of said first carbonate polymer whereby the additive composition in said polymer/concentrate blend is present in an amount effective to render said blend ignition resistant; and

C) pelletizing or molding said carbonate polymer/concentrate blend.

The advantages of the invention over the known techniques are that significant improvement is seen in the toughness of the final product as measured by improved Izod impact tests when the polymer concentrate is first made and diluted down with additional polymer. In addition, the generation of dust particles which is commonly found in additive compounding is eliminated.

The carbonate polymers employed in the present invention are advantageously aromatic carbonate polymers such as the trityl diol carbonates described in U.S. Patent Nos. 3,036,036; 3,036,037; 3,036,038 and 3,036,039; polycarbonates of bis(ar-hydroxyphenyl) alkylidenes (often called bisphenol-A type diols) including their aromatically and aliphatically substituted derivatives such as disclosed in U.S. Patent Nos. 2,999,835; 3,038,365 and 3,334,154; and carbonate polymers derived from other aromatic diols such as described in U.S. Patent No. 3,169,121.

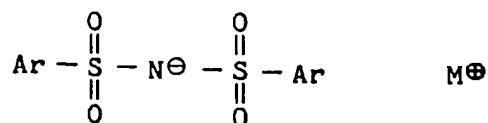
It is understood, of course, that the polycarbonate may be derived from (1) two or more different dihydric phenols or (2) a dihydric phenol and a glycol or a hydroxy- or acid-terminated polyester or a dibasic acid in the event a carbonate copolymer or heteropolymer rather than a homopolymer is desired. Also suitable for the practice of this invention are blends of any one of the above carbonate polymers. Also included in the term carbonate polymer are the ester/carbonate copolymers of the types described in U.S. Patent Nos. 3,169,121; 4,105,633; 4,156,069; 4,225,556; 4,260,731;

4,287,787; 4,330,662; 4,355,150 4,360,656; 4,374,973; and 4,388,455. Of the aforementioned carbonate polymers, the polycarbonates of bisphenol-A and derivatives, including copolycarbonates of bisphenol-A, are preferred. Methods for preparing carbonate polymers for use in the practice of this invention are well known. For example, several suitable methods are disclosed in the aforementioned patents.

5 The monomeric, oligomeric or polymeric halogenated aromatic compound used in this invention can be virtually any halogenated organic compound commonly used as a fire retardant additive. The preferred compounds are the halo-substituted aromatic compounds (halo is fluoro, chloro, or bromo). Suitable compounds include, for example, decabromo diphenyloxide, tris(tribromophenoxy) triazine, decabromodiphenylcarbonate, an oligomer or polymer of tetrabromobisphenol A, and a copolymer of bisphenol A/tetrabromobisphenol A. Combinations of the above identified compounds can be employed. Examples of other suitable monomeric and polymeric halogenated compounds are disclosed in U.S. Patent No. 4,263,201.

10 The metal salts of sulfur compounds used herein include metal salts of aromatic sulfonates, sulfates, sulfonamides, and sulfimides. Suitable metals are the metals of Groups I and IIA of the Periodic Chart as well as copper, aluminum, and antimony. The preferred metal is an alkali metal such as sodium or potassium.

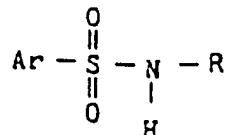
15 The preferred group of aromatic sulfur compounds are sulfimides having the formula



20 wherein Ar is an aromatic group and M is a metal cation.

25 Examples of the sulfimide salts are the alkali metal salts of saccharin, N-(p-tolylsulfonyl)-p-toluene sulfimide, N-(N'-benzylaminocarbonyl)sulfanilimide, N-(phenylcarboxyl)-sulfanilimide, N-(2-pyrimidinyl)-sulfanilimide, and N-(2-thiazolyl)sulfanilimide. These salts and similar ones are disclosed in U.S. Patent No. 4,254,015.

The free aromatic sulfimides useful in this invention are those having a pKa in the range of from 1 to 3. Examples of such free aromatic sulfimides are saccharin, N-(p-tolylsulfonyl)-p-toluene sulfimide, N-(N'-benzylaminocarbonyl)sulfanilimide, N-(phenylcarboxyl)-sulfanilimide, N-(2-pyrimidinyl)-sulfanilimide, and N-(2-thiazolyl)sulfanilimide. They are further illustrated by the formula



30 35 wherein R is carbonyl, arylcarbonyl, arylaminocarbonyl, aralkylaminocarbonyl, or arylsulfonyl. Specific examples of these groups are benzoyl, benzylaminocarbonyl and tolylsulfonyl groups.

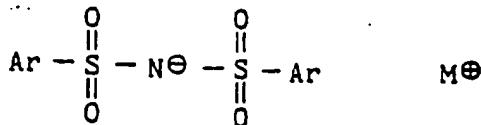
In general, the additive package has a fixed weight ratio of components so that after the polymer concentrate is made it can be readily shipped without dust formation to desired locations where it can be diluted down or let down with more polymer to the final ignition resistant product. The preferred ratio of the components, metal salt of an aromatic sulfur containing compound : monomeric, oligomeric or polymeric halogenated aromatic compound : metal salt of an inorganic compound : fibril-forming polytetrafluoroethylene is 1:10:1:3. For example, a 15 percent additive/polymer concentrate is blended with more polymer at a 10:1 ratio to give a useful ignition resistant blend.

In addition to the aforementioned fire retardant additives, other additives can be included in the carbonate polymer composition of the present invention such as fillers (i.e. glass fibers), pigments, dyes, antioxidants, stabilizers, ultraviolet light absorbers, mold release agents, impact modifiers and other additives commonly employed in carbonate polymer compositions.

The following examples and controls are presented to further illustrate the invention.

50 Example 1

A fifteen percent ignition resistant (IR) additive/ polycarbonate concentrate was prepared by adding 1 gram (1 percent) potassium paratolylsulfimide (KPTSM) 1 gram (1 percent) potassium bisulfate (KHSO_4), 3 grams (3 percent) fibril-forming polytetrafluoroethylene (Teflon 6C) and 10 grams (10 percent) tetrabromobisphenol-A oligomer (TBBPA), all in one masterbatch, to 85 gms (85 percent) heat stabilized polycarbonate resin polycarbonate carrier having a 22 gms/10 min MFR (melt flow rate). The KPTSM is represented by the formula:



wherein Ar is a paratolyl group and M is potassium

The additive masterbatch and polycarbonate pellets were blended on a rotating blender (Lightnin blender) for one minute with agitation. The resultant blended material was gradually fed to a 30 mm twin screw extruder having a 250°C barrel temperature. The extruded pellets were post blended on a paint shaker to ensure uniform mixing.

10 The 15 percent IR concentrate was let down in or blended with a base polycarbonate at a 10 to 1 ratio. The base polycarbonate had a 15 gms/10 minute melt flow rate (MFR). The mixture was blended on Lightnin blender for 1 minute. The blended pellets were extruded on 30 mm twin screw extruder at 275°C barrel temperature to make a 15 MFR ignition resistant polycarbonate product. The final product was dried in an oven for 3 hours at 125°C and molded into bars for 15 flammability and Izod impact tests. If desired, the IR concentrate and base polycarbonate can be fed separately into the twin screen extruder or can be directly molded into various parts.

Example 2

20 The procedure for Example 1 was repeated using 1 gm (1 percent) paratolylsulfimide (HPTSM) in place of the KHSO₄.

Example 3

25 The procedure for Example 1 was repeated using a 30 weight percent IR concentrate. This concentrate contained

2 percent potassium paratolylsulfimide,
 2 percent potassium bisulfate,
 6 percent fibril-forming polytetrafluoroethylene and
 30 20 percent tetrabromobisphenol-A oligomer.

This 30 percent concentrate was blended with a base polycarbonate at a 20 to 1 letdown ratio.

Example 4

35 The procedure for Example 3 was repeated using 2 gm (2 percent) paratolylsulfimide (HPTSM) in place of the 2 percent potassium bisulfate (KHSO₄).

Control A

40 The procedure for Example 1 was repeated using only the base resin without either IR concentrate or IR masterbatch.

Control B

45 The procedure for Example 1 was repeated using only 1.5 percent IR additive masterbatch without concentrate.

Bars obtained in the above examples and controls were tested for Izod impact strength and the results are shown in Table I.

Table I

50

Sample	PC carrier (MFR in gms/10 min.)	Izod Impact 10 mil (0.254 mm) notched at 25°C; ASTM D-259-84	
		Izod Impact ft. lb/in (J/m)	% Brittle
Cntrl A (Base Polymer)	N.A.	15.0(800)	0
Cntrl B (Cntrl A + 1.5% IR Masterbatch)	N.A.	3.0(160)	100

55 Continuation of the Table on the next page

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Table I (continued)

Sample	PC carrier (MFR in gms/10 min.)	Izod Impact 10 mil (0.254 mm) notched at 25°C; ASTM D-259-84	
		Izod Impact ft. lb/in (J/m)	% Brittle
Example 1 15% I.R. concentrate with 10:1 letdown	22	14.5(775)	0
Example 2 15% I.R. concentrate with 10:1 letdown	22	14.7(785)	0
Example 3 30% I.R. concentrate with 20:1 letdown	22	13.0(694)	10
Example 4 30% I.R. concentrate with 20:1 letdown	22	13.3(710)	10

Table I shows that the use of IR concentrate at either 10 to 1 or 20 to 1 letdown (15 or 30 percent) resulted in improved Izod impact properties without any brittle breaks versus IR additive masterbatch which resulted in very low Izod impact with 100 percent brittle breaks. The improvement of Izod impact via the use of IR concentrate is attributed to more uniform additive dispersion using IR concentrate than using IR masterbatch.

Bars obtained in the above examples and controls were further tested for ignition resistance or flammability using the well known UL-94 test. The results are shown in Table II.

Table II

UL-94 Test, 1/16" (1.6 mm)

<u>Sample</u>	<u>Avg. T-Sec</u>	<u>Number of Drips</u>	<u>Rating</u>
Control A	10.3	5/5	V-2
Control B	1.0	0/5	V-0
Example 1	1.2	0/5	V-0
Example 2	1.5	0/5	V-0
Example 3	1.1	0/5	V-0
Example 4	1.3	0/5	V-0

Note: T-Sec means time in seconds for a flame out

Table II shows the use of IR concentrate does not detract from the flammability properties of final product.

Example 5

The procedure for Example 3 was repeated using a 20 MFR polycarbonate carrier.

Example 6

The procedure for Example 4 was repeated using a 15 MFR polycarbonate carrier.

Example 7

The procedure for Example 4 was repeated using a 10 MFR polycarbonate carrier.

Control C

The procedure for Example 3 was repeated using a 40 MFR polycarbonate carrier.

5 Control D

The procedure for Example 3 was repeated using a 80 MFR polycarbonate carrier.

Bars obtained in the above examples and controls were tested for Izod impact strength and the results are shown in Table III.

10

Table III

15	Sample	PC Carrier (MFR)	Izod Impact, 10 mil (0.254 mm) notched at 25°C; ASTM D-259-84	
			Izod Impact (ft.lb./in)	% Brittle
20	Cntrl A (Base Polymer)	N.A.	15.0(800)	0
	Cntrl C (Cntrl A + 30% IR concentrate at 20 to 1 ratio)	40	7.5(400)	50
	Cntrl D (Cntrl A + 30% IR concentrate at 20 to 1 ratio)	80	3.0(160)	100
25	Example 5 (Cntrl A + 30% IR concentrate at 20 to 1 ratio)	20	13.0(694)	0
	Example 6 (Cntrl A + 30% IR concentrate at 20 to 1 ratio)	15	14.5(775)	0
	Example 7 (Cntrl A + 30% IR concentrate at 20 to 1 ratio)	10	14.0(747)	10

30 The above controls and/or examples show that high melt flow rate polycarbonates are not effective as carriers as the low MFR carriers.

Bars obtained in the above examples and controls were further tested for ignition resistance using the UL-94 test. The results are shown in Table IV.

35

Table IV

UL-94 Test, 1/16"
(1.6 mm)

40

	<u>Sample</u>	<u>Avg. T-Sec</u>	<u>Number of Drips</u>	<u>Rating</u>
45	Control A	10.3	5/5	V-2
	Control C	1.0	0/5	V-0
	Control D	2.0	0/5	V-0
50	Example 5	1.0	0/5	V-0
	Example 6	1.0	0/5	V-0
	Example 7	1.0	0/5	V-0

55

Note: T-Sec means time in seconds for a flame out

Tables III and IV show that lower MFR polycarbonate carriers help to improve the Izod impact strength of the polymer

better than higher MFR PC carriers without sacrificing the flammability property of the final product.

Claims

- 5 1. A process for dispersing powdered ignition resistant additives into carbonate polymers to make polymers having an embrittlement of less than 50% which comprises
 - A) preparing a base concentrate by melt blending a carbonate polymer having a melt flow rate in the range of from about 3 to less than 40 with from 5 to 50 weight percent based on concentrate weight of one or more powdered ignition resistant additives and including one or more of the flame retardant additives selected from the group of:
 - i) a metal salt of an organic aromatic sulfur containing compound, ii) a monomeric, oligomeric or polymeric halogenated aromatic compound,
 - iii) a metal salt of an inorganic compound
 - iv) a free aromatic sulfimide, and
 - v) a fibril forming polytetrafluoroethylene,
 - 20 B) pelletizing said base concentrate;
 - C) blending said concentrate pellets with carbonate polymer pellets having a melt flow rate from 1 to less than 40 whereby the additive composition in said polymer/concentrate blend is present in an amount effective to render said blend ignition resistant; and
 - 25 D) pelletizing or molding said carbonate polymer/concentrate blend.
- 30 2. A process as claimed in Claim 1 wherein the additives consist of a mixture of a metal salt of an aromatic sulfur containing compound, a monomeric, oligomeric or polymeric halogenated aromatic compound, a metal salt of an inorganic compound or a free aromatic sulfimide, and a fibril-forming polytetrafluoroethylene.
- 35 3. A process as claimed in Claim 2 wherein the additives consist of an alkali metal salt of paratolylsulfimide, tetrabromobisphenol-A oligomer, an alkali metal sulfate salt and fibril-forming polytetrafluoroethylene.
- 40 4. A process as claimed in Claim 1 wherein the polycarbonate polymer in step A) and step C) is a bisphenol A polycarbonate and the additives comprise:
 - i) from 2.0 to 10 percent by weight of a metal salt of an aromatic sulfur-containing compound,
 - ii) from 10 to 80 percent by weight of a monomeric, oligomeric or polymeric halogenated aromatic compound,
 - iii) from 2.0 to 10.0 percent by weight of compound selected from a metal salt of an inorganic compound and a free aromatic sulfimide, and
 - 45 iv) from 6 to 40 percent by weight of a fibril-forming polytetrafluoroethylene.
- 50 5. A process as claimed in Claim 1 wherein in step A) a carbonate polymer having a melt flow rate of from 3 to 20 is dry blended with from 5 to 50 weight percent of said additives.
- 55 6. A process as claimed in Claim 5 wherein the polycarbonate polymer in step A) is a polycarbonate resin homopolymer made from bisphenol A and has a melt flow rate of from 3 to 20.
7. A process as claimed in Claim 4 wherein said aromatic sulfur-containing compound is an aromatic sulfonate, aromatic sulfate, aromatic sulfonamide or aromatic sulfimide.
8. A process as claimed in Claim 7 wherein the aromatic sulfimide additive consists of an alkali metal salt of paratolyl-sulfimide.

9. A process as claimed in Claim 4 wherein said halogenated aromatic compound is tetrabromobisphenol-A oligomer.
10. A process as claimed in Claim 4 wherein said metal salt of an inorganic compound is potassium bisulfate and said free aromatic sulfimide is paratolylsulfimide.
- 5 11. The process of Claim 1 wherein the powdered ignition resistant or flame retardant additives comprise a metal salt of an aromatic sulfur containing compound and a fibril forming polytetrafluoroethylene.

10 **Patentansprüche**

1. Verfahren zum Dispergieren von gepulverten entzündungsfesten Zusatzstoffen in Carbonatpolymeren zur Herstellung von Polymeren mit einer Versprödung von weniger als 50 %, welches umfaßt:
 - 15 A) Herstellung eines Grundkonzentrates durch Schmelzmaschen eines Carbonatpolymeren, das eine Fließfähigkeit von etwa 3 bis weniger als 40 besitzt, mit von 5 bis 50 Gew.-%, bezogen auf Konzentratgewicht, eines oder mehrerer gepulverter entzündungsfester Zusatzstoffe, der/die einen oder mehrere der aus der folgenden Gruppe ausgewählten Zusatzstoffe einschließen:
 - 20 i) ein Metallsalz einer organischen aromatischen schwefelhaltigen Verbindung,
 - ii) eine monomere, oligomere oder polymere halogenierte aromatische Verbindung,
 - iii) ein Metallsalz einer anorganischen Verbindung,
 - iv) ein freies aromatisches Sulfimid, und
 - v) ein fibrillenbildendes Polytetrafluoroethylen,
 - 25 B) Pelletisieren dieses Grundkonzentrates,
 - C) Mischen dieser Konzentratpellets mit Carbonatpolymerpellets mit einer Fließfähigkeit von 1 bis weniger als 40, wodurch die Zusatzstoffzusammensetzung in dieser Polymer/Konzentrat-Mischung in einer effektiven Menge, um diese Mischung entzündungsfest zu machen, vorhanden ist, und
 - 30 D) Pelletisieren oder Verformen dieser Carbonatpolymer/Konzentrat-Mischung.
2. Verfahren nach Anspruch 1, worin die Zusatzstoffe aus einer Mischung eines Metallsalzes einer aromatischen schwefelhaltigen Verbindung, einer monomeren, oligomeren oder polymeren halogenierten aromatischen Verbindung, eines Metallsalzes einer anorganischen Verbindung oder eines freien aromatischen Sulfimids und einem fibrillenbildenden Polytetrafluoroethylen bestehen.
3. Verfahren nach Anspruch 2, worin die Zusatzstoffe aus einem Alkalimetallsalz von para-Tolylsulfimid, Tetrabrombisphenol-A-oligomerem, einem Alkalimetallsulfatsalz und einem fibrillenbildenden Polytetrafluoroethylen bestehen.
4. Verfahren nach Anspruch 1, worin das Polycarbonatpolymere in Stufe A) und Stufe C) ein Bisphenol-A-polycarbonat ist und die Zusatzstoffe umfassen:
 - 45 i) von 2,0 bis 10 Gew.-% eines Metallsalzes einer aromatischen schwefelhaltigen Verbindung,
 - ii) von 10 bis 80 Gew.-% einer monomeren, oligomeren oder polymeren halogenierten organischen Verbindung,
 - iii) von 2,0 bis 10 Gew.-% einer Verbindung, ausgewählt aus einem Metallsalz einer anorganischen Verbindung und einem freien aromatischen Sulfimid, und
 - iv) von 6 bis 40 Gew.-% eines fibrillenbildenden Polytetrafluoroethylen.
5. Verfahren nach Anspruch 1, worin in Stufe A) ein Carbonatpolymeres, das eine Fließfähigkeit von 3 bis 20 besitzt, mit von 5 bis 50 Gew.-% dieser Zusatzstoffe trockengemischt wird.
6. Verfahren nach Anspruch 5, worin das Polycarbonatpolymere in Stufe A) ein Polycarbonatharzhomopolymeres ist, das aus Bisphenol-A hergestellt wurde und eine Fließfähigkeit von 3 bis 20 besitzt.
- 55 7. Verfahren nach Anspruch 4, worin diese aromatische schwefelhaltige Verbindung ein aromatisches Sulfonat, aromatisches Sulfat, aromatisches Sulfonamid oder aromatisches Sulfimid ist.
8. Verfahren nach Anspruch 7, worin der aromatische Sulfimid-Zusatzstoff aus einem Alkalimetallsalz von para-Tolyl-

sulfimid besteht.

9. Verfahren nach Anspruch 4, worin diese halogenierte aromatische Verbindung Tetrabrombisphenol-A-oligomeres ist.

5 10. Verfahren nach Anspruch 4, worin dieses Metallsalz einer anorganischen Verbindung Kaliumbisulfat ist und dieses freie aromatische Sulfimid para-Tolylsulfimid ist.

10 11. Verfahren nach Anspruch 1, worin die gepulverten entzündungsfesten oder flammhemmenden Zusatzstoffe ein Metallsalz einer aromatischen schwefelhaltigen Verbindung und ein fibrillenbildendes Polytetrafluorethylen umfassen.

Revendications

15 1. Procédé permettant de disperser des adjuvants d'ignifugation en poudre dans des polycarbonates de façon à obtenir des polymères présentant un taux de fragilisation inférieur à 50 %, lequel procédé comportant les étapes consistant à :

20 A) préparer un concentré de base en mélangeant à l'état fondu un polycarbonate dont l'indice de fluidité à chaud vaut d'environ 3 à moins de 40, avec de 5 à 50 % en poids, par rapport au concentré, d'un ou plusieurs adjuvants d'ignifugation en poudre, comprenant un ou plusieurs des adjuvants d'ignifugation choisis dans le groupe constitué par :

- 25 1) un sel métallique d'un composé organique aromatique soufré,
 2) un composé aromatique halogéné monomère, oligomère ou polymère,
 3) un sel métallique d'un composé minéral,
 4) un sulfimide aromatique libre, et
 5) un polytétrafluoroéthylène formant des fibrilles ;

30 B) mettre sous forme de pastilles ledit concentré de base ;
 C) mélanger lesdites pastilles de concentré avec des pastilles de polycarbonate dont l'indice de fluidité à chaud vaut de 1 à moins de 40, la composition d'adjuvants se trouvant, dans ledit mélange de polymère et de concentré, en une quantité qui rend effectivement ledit mélange ignifugé ; et
 D) mettre sous forme de pastilles ou mouler ledit mélange de polycarbonate et de concentré.

35 2. Procédé conforme à la revendication 1, dans lequel les adjuvants sont constitués d'un mélange d'un sel métallique d'un composé aromatique soufré, d'un composé aromatique halogéné monomère, oligomère ou polymère, d'un sel métallique d'un composé minéral ou d'un sulfimide aromatique libre, et d'un polytétrafluoroéthylène formant des fibrilles.

40 3. Procédé conforme à la revendication 2, dans lequel les adjuvants sont constitués d'un sel de métal alcalin de para-tolylsulfimide, d'un oligomère à base de tetrabromobisphénol A, d'un sel qui est un sulfate de métal alcalin, et d'un polytétrafluoroéthylène formant des fibrilles.

45 4. Procédé conforme à la revendication 1, dans lequel le polymère de type polycarbonate utilisé dans les étapes A) et C) est un polycarbonate à base de bisphénol A, et les adjuvants comprennent :

- 50 1) de 2,0 à 10 % en poids d'un sel métallique d'un composé organique aromatique soufré,
 2) de 10 à 80 % en poids d'un composé aromatique halogéné monomère, oligomère ou polymère,
 3) de 2,0 à 10,0 % en poids d'un composé choisi parmi un sel métallique d'un composé minéral et un sulfimide aromatique libre, et
 4) de 6 à 40 % en poids d'un polytétrafluoroéthylène formant des fibrilles.

55 5. Procédé conforme à la revendication 1, dans lequel, dans l'étape A), on mélange à sec un polycarbonate dont l'indice de fluidité à chaud vaut de 3 à 20 avec de 5 à 50 % en poids desdits adjuvants.

6. Procédé conforme à la revendication 5, dans lequel le polycarbonate utilisé dans l'étape A) est une résine homo-

polymère de polycarbonate obtenue à partir de bisphénol A, dont l'indice de fluidité à chaud vaut de 3 à 20.

7. Procédé conforme à la revendication 4, dans lequel ledit composé aromatique soufré est un sulfonate aromatique, un sulfate aromatique, un sulfonamide aromatique ou un sulfimide aromatique.
- 5 8. Procédé conforme à la revendication 7, dans lequel l'adjuvant de type sulfimide aromatique est un sel de métal alcalin de paratolylsulfimide.
- 10 9. Procédé conforme à la revendication 4, dans lequel ledit composé aromatique halogéné est un oligomère à base de tétrabromobisphénol A.
- 10 10. Procédé conforme à la revendication 4, dans lequel ledit sel métallique d'un composé minéral est du bisulfate de potassium, et ledit sulfimide aromatique libre est du paratolylsulfimide.
- 15 11. Procédé conforme à la revendication 1, dans lequel les adjuvants d'ignifugation en poudre comprennent un sel métallique d'un composé aromatique soufré et un polytétrafluoroéthylène formant des fibrilles.

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